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CEMENT AND LIME MANUFACTURE

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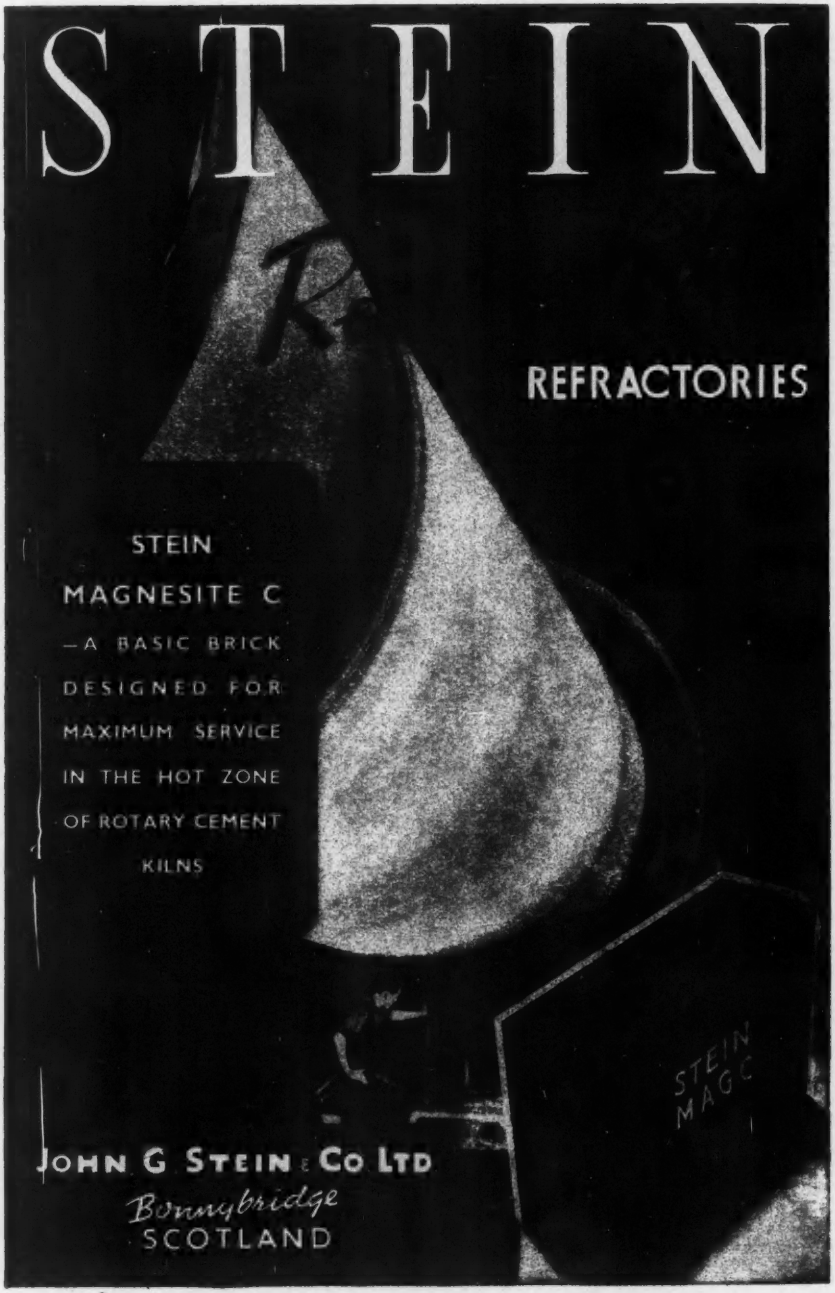
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Froth Flotation in Cement Manufacture.

IN this journal for July, 1940, the froth-flotation process in the manufacture of cement is briefly described. In the following are given more details of the principles of the process and the reagents used.

When the raw materials available are unsuitable for the production of high quality cement by ordinary methods consideration may be given to the use of the flotation process. By this process the raw materials can be refined and controlled so that Portland cement of the highest quality can be made, and in addition it is possible to meet the requirements of modified cements, including low-heat and sulphate-resistant cements, by making relatively simple adjustments. Examination of the raw material resources and quarry development schemes may be possible by the specific application of the flotation process since materials unsuitable for the ordinary process of manufacture may be found to be usable economically. The economics of the process require very careful consideration, since the initial cost of the processing and auxiliary plant and the operation costs are high relative to the cost of modern closed-circuit wet-grinding systems. An item which should not be overlooked is the possibility of the recovery of useful industrial minerals from the rejects. For example, at a works in Finland calcite is separated from Wollastonite and the latter is sold for glass manufacture.

The complete process comprises a combination of grinding, classification, flotation, and thickening methods, each adapted to satisfy the requirements of cement and the characteristics of calcareous and argillaceous materials. Extremely small quantities of organic chemicals are used which cause certain of the finely-ground minerals to float and others to sink in water, whereby the undesirable minerals, or excessive proportions of useful minerals, can be separated and discarded.

Several years ago Mr. C. H. Breerwood, of the Valley Forge Cement Co.* of the U.S.A., found that, even though the chemical analysis of the raw-material mixture

* The flotation method used at the works of the Valley Forge Cement Co. are described in this journal, July, 1940.

remained constant, the cements produced varied widely in quality. His investigations revealed that silica in the form of quartz did not react to form the essential calcium silicates if the quartz particles were retained on a No. 325 U.S. sieve. Experiments were made with the flotation process, the material being suspended in about four times its weight of water and agitated in a flotation cell in which was placed a small quantity of a fatty acid. The fatty acid forms a film of insoluble metallic soap on the surface of the calcite grains, making them repel water and to be subject to the attachment of air bubbles which makes them float to the surface. The other minerals, not being filmed, sink in the flotation cell. This made it possible to remove the quartz. In order to fulfil the requirements of low-heat and sulphate-resistant cement specifications, the alumina content should be limited ; its common mineral forms in cement materials are mica, kaolin and feldspar. Reagents have been adapted to the flotation process which cause these minerals to float away from the calcite and quartz, both of which are recovered. Both types of flotation are being used either alone or in combination in order to fulfil the chemical requirements of the special types of cement. At the Valley Forge plant compositions for all known types of Portland cement can be produced solely from the argillaceous limestone quarried on the site.

Flotation Reagents.

The reagents may be divided into four main groups according to their function, namely, (1) Frothers ; (2) Collecting reagents or promoters ; (3) Dispersers, and (4) Depressors. New reagents are being produced and patented and the following list is by no means intended to be comprehensive.

FROTHERS.—Typical materials are mixtures of monohydric alcohols, dilute resinates, and cresylic acid. The quantities required are small and are introduced at various points in the circuit to maintain the desired froth balance. The major portion of the water of the froth is returned to the flotation circuit from the thickener, which accounts in part for the economy in consumption.

COLLECTORS OR PROMOTERS.—(a) For calcite flotation. The usual reagents are unsaturated fatty acids, resin acids, and their soaps and emulsions. Collectors in common use are oleic acid, fish oil, fatty acids and their emulsions and soaps. Talloel, a mixture of fatty and resin acids derived from the black liquor soap of the sulphate process for the manufacture of paper, and soaps and emulsions thereof, are effective collectors of calcite. Some soaps which are used for certain argillaceous limestone slurries have definite frothing characteristics; these collectors are generally saponified fatty and resin acids. In general the emulsions are more satisfactory than the soaps in the flotation of calcite, since, in the differential separation of this mineral, soaps sometimes create an excessive and comparatively barren froth. Resin acids are generally inferior collectors, and unmodified fatty acids of high liter are generally unsatisfactory for use in the usual argillaceous limestone slurries. The successful concentration of calcite from the siliceous minerals in finely divided slurries is principally the result of adding the collecting reagent in extremely small quantities in each cell of the circuit.

(b) For siliceous flotation. These reagents are commonly known as " cationic " reagents. They may be described as chemicals which give, in solution, the long-chain surface-active group in the positive ion, and in which the negative ion is generally a halogen. One of this class of reagents specifically mentioned as being in use at the Valley Forge plant has dodecylamine hydrochloride as its principal constituent. Many of the cationic reagents have a critical *pH* range and are ineffective in alkaline slurries, the normal range in which they are effective being from *pH* 7.4 to *pH* 8. These reagents modify the surfaces of the siliceous mineral grains with limited filming of the oxides. Mr. Breerwood found that the reagents would float all or substantially all of the mineral constituents, including the acidic, oxide, and sulphide minerals present in argillaceous limestone slurries but that by carefully limiting the quantity and by introducing very small increments at each step differential separation can be made, silicates being readily separated from silica and calcite. Also, micas and talc may be separated as a concentrate to be discarded, and to recover the calcite, quartz, and composite rock particles as a tailing of flotation.

DISPERSERS.—The most effective reagents of this class are calcium lignin sulphonate either alone or with the addition of sodium silicate or soda ash, and are employed in finely-divided argillaceous limestone slurries to reduce natural flocculation and colloidal filming.

DEPRESSORS.—These are used to decrease floatability of an undesired mineral. Thus calcium lignin sulphonate, effective as a disperser, also depresses carbon in various mineral slurries.

Theories and Physical Action of Flotation.

There are two theories on the action of collecting reagents on the surfaces of the mineral grains on which they concentrate. The chemical theory assumes a chemical reaction (a metathesis) between the avion of a fatty acid, for example, and the hydrolysed surface of the oxide ore mineral to produce an oriented monomolecular film of insoluble metallic soap. The absorption theory supposes a surface tension difference at the solid-liquid interface, in which a surface-active part of the collector unites with the hydrolysed surface of the mineral grain. In any case, the ore mineral particle is at least partially filmed, which makes the surface water-repellent and accordingly subject to air-bubble attachment.

The mechanics of froth flotation comprise air-bubble attachment to water-repellent surfaces of the ore minerals. These bubbles are considered to be re-formed from solution at the water-solid interfaces. In sulphide flotation the air bubbles are said to be relatively large, the ore particles adhering to them and being floated to the surface of the slurry. At the surface the particles and bubbles attach themselves to the froth bubbles, are stabilised by the frothing agent, and overflow as the concentrate. In the concentration of calcite from argillaceous limestones the mechanics are quite different, particularly because of the extreme fineness of the particles and the nature of air.bubble attachment. The air bubbles attached to the calcite grains are so fine that they cannot be seen owing to movements

attributable to surface tension. A number of grains of calcite may be seen bound together in groups in the form of air bubble-mineral floccules, in which the particles are each surrounded and bonded by air bubbles in an increasing order of sizes from the grain surfaces. The air entrained in each floccule causes it to rise and attach itself to a large froth bubble on the surface of the cell.

Chemical-Resistant Resin Cements.

THE development and use in America of phenol-formaldehyde-resin cements, and furan-resin cements, which are resistant to chemical attack, are described in a recent number of "Industrial and Engineering Chemistry." These cements supplement the earlier types of chemical-resistant cements such as a sodium-silicate cement and sulphur cement.

Phenolic-resin cements are resistant to most acids that are not highly oxidizing, solvents, weak alkalis, steam at 360 deg. F., fats, and oils. They have a tensile strength of 1,000 lb. per square inch and a compressive strength of 10,000 lb. per square inch, and are of low porosity. Liquid phenolic resin containing about 75 per cent. of solids is mixed at the time of using with a silica or carbon filler containing a hardening agent, which produces controlled but rapid setting of the liquid resin at atmospheric temperature.

Furan-resin cement, which has about the same physical properties as phenolic-resin cement, is a more recent product. It is liquid furan-resin mixed at the time of using with crushed silica or carbon containing a hardening agent. The mixture sets within an hour at ordinary temperatures. It is resistant to many acids, solvents, and alkalis of any concentration.

Mortars of phenolic-resin and furan-resin cements are used for jointing brick linings of chemical storage tanks and brick and tiled floors in food-processing works. Reinforced concrete and metal tanks containing virulent liquids are lined with impervious acid-resistant asphalt, plastic sulphur-cement, synthetic resins, and similar materials. This lining is protected by a corrosion-resistant brick lining set in a suitably resistant cement mortar. The brick lining also serves as a thermal insulator to the inner lining, and may also protect it from corrosive attack. A drop of 45 deg. F. is common with 4 in. of acid-resistant brick. The choice of material for the brick lining depends upon the nature and temperature of the chemicals to be stored, and the thickness depends on the temperature, size of the tank, and vibration or other disturbances due to the equipment. No corrosion-resistant cement can withstand all chemicals, but it is claimed that one of the four types, that is sodium-silicate, sulphur, phenolic-resin, and furan-resin cements, is suitable for a particular chemical or combination of chemicals.

The Transport of Loose Cement.

THE following is an abstract of an article by E. Plassmann, E. Ruhland, and O. Färber, of the Dyckerhoff Portland Cement Works, published in "Zement-Kalk-Gips". The authors deal with the transport of loose cement in the light of practical experience of various methods. In their opinion the paper bag is, and will remain, the best means of packing and transporting cement, and if it were not for shortage of paper there would be no need to consider other methods.

Rail Transport.

The covered goods wagon is commonly employed for carrying loose cement. Before a wagon is put to this use the floor and walls should be tested for tightness, and open joints covered or stopped. Door openings may be blocked by stacking filled cement bags across them so as to procure an enclosed space into which loose cement may be discharged by means of a movable steel pipe or flexible rubber hose. A suitable method of conveying the material from the storage bins to the wagon is illustrated in *Fig.* 1, where (a) is a worm conveyer from the storage

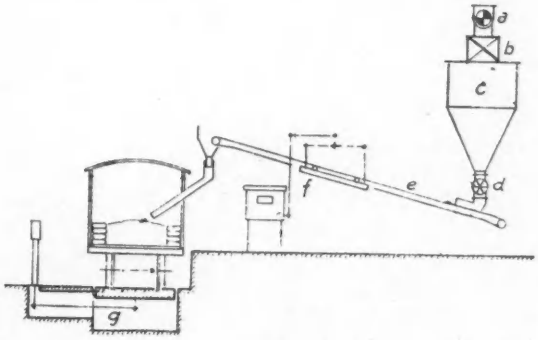

Fig. 1.

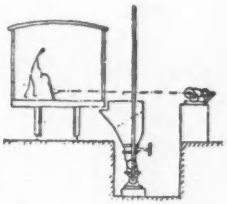

Fig. 2.

bins, (b) is an automatic gauge box, (c) a measuring hopper, (d) an automatic outlet valve for determining the volume from the number of revolutions multiplied by the volume of the valve, (e) a conveyer belt, (f) an automatic weighing machine with a cut-off for predetermined quantities, and (g) a weighbridge to determine the weight of empty and laden wagons; alternatively a weighing machine may be installed on the conveyer belt. In the case of small quantities only one of the measuring devices shown in *Fig.* 1 may be necessary.

To empty the wagon, the doors are opened and a sufficient number of bags removed to provide a working space from which the loose material can be discharged by hand, by power shovel, by a cement pump, or where suitable (for example, when unloading from barges) the grab of an excavator may be used. When the wagons are unloaded by hand the cement may be shovelled from the

wagon on to a portable conveyer which takes it to storage or to another vehicle, or it may be shovelled directly into a bin erected beside the wagon and the top of which is below the level of the floor of the wagon, or it may be shovelled into the boot of a bucket elevator which takes it to an elevated storage bin. When a cement pump is used the wagon may be emptied by a mechanical shovel into

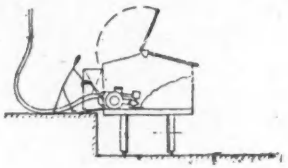

Fig. 3.

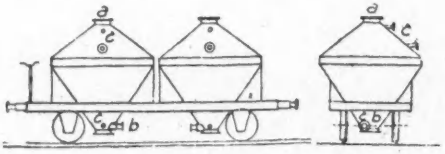

Fig. 4.

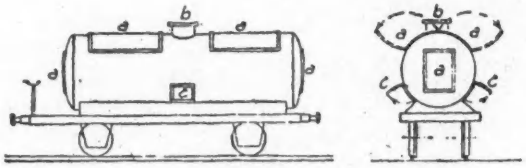

Fig. 5.

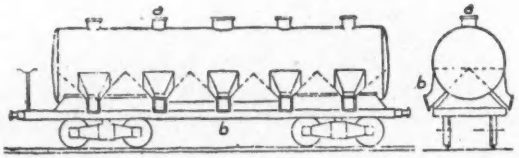

Fig. 6.

the hopper of the pump (*Fig.* 2), or a portable pump may be placed inside the wagon (*Fig.* 3).

An open goods wagon is suitable only for short distances and during fine weather, when the cement can be sufficiently protected against atmospheric influences by the provision of a suitable covering. Of the special container wagons available, the vertical hopper-type is particularly suitable. A wagon with two containers of a combined capacity of 26 tons is shown in *Fig.* 4, where the inlets are shown at (a), the connections for the emptying pipes at (b), and the connections for the cement pump at (c). The openings are secured against wind and rain by tight-fitting covers. The hoppers are emptied through delivery pipes of 4 in. to 6 in. diameter connected to the outlets, and conveyed through pipes to storage bins which may be at a horizontal distance of up to 165 ft. and a height of up to 33 ft. above rail level. Compressed air introduced into the hopper through any of the connections provided for this purpose has the effect of loosening

the cement. Air collects at the top of the container and its pressure forces the cement through the pipe into the storage bins. The bin is fitted with a filter hose or flue, so as to allow the escape of any surplus air that may collect in the interior of the bin. The advantages of this method are its simplicity, the absence of dust, and the fact that no manual labour is required.

A 2-axle flat-bottomed wagon carrying a container of 24 tons capacity is illustrated in *Fig.* 5, and a container of 38 to 45 tons capacity on a 2-bogie truck is shown in *Fig.* 6. The container in *Fig.* 5 has at (a) large openings at the top and ends suitable also for bulky goods, (b) a manhole for granular material, and (c) an outlet for granular material. The container shown in *Fig.* 6 has at (a) additional inlet openings and at (b) additional outlet openings. Compared with the hopper-type container, the disadvantage of the boiler-type tank is that compressed air cannot be used when emptying it. The contents of the tank are loosened and stirred by rodding, causing the cement to move towards the outlets

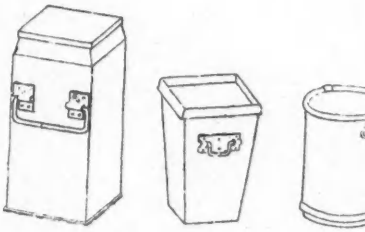

Fig. 7.

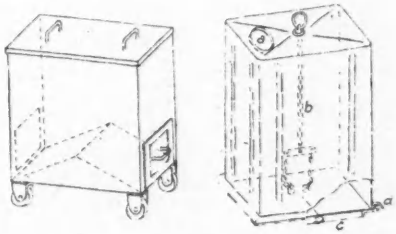

Fig. 8.

at the bottom. To dispose of any residue in the container, men clean it out by hand with shovels.

Transport by Road.

Loose cement is at present generally transported in flat-bottomed open lorries. During transport the vibration of the vehicle causes compaction of the cement, with the result that losses due to wind are very slight, amounting to no more than about 1 per cent. If desired, the lorry can be provided with a covering. Unless the lorry is of the tipping type, an inclined chute is necessary to unload the cement. Closed containers mounted on the lorries are more efficient. Containers in use with capacities of about 1 cwt. and 2 cwt. are shown in *Fig.* 7. They may be provided with loose covers, or lids fitted with locking devices. The use of an automatic weighing machine is particularly suitable so as to ensure that the containers receive accurate quantities. Containers of 1 to 2 tons capacity are shown in *Fig.* 8.

Special lorries with vertical hoppers, similar to those for railway wagons illustrated in *Fig.* 4, are extremely serviceable. A supply of compressed air required for emptying the tank is obtained from a compressor coupled to the engine of the vehicle.

A further development is the use of horizontal barrel containers on flat-bottomed lorries. A container of this type is shown in *Fig.* 9, where (a) are the inlet and outlet openings, (b) the barrel hoops, (c) the coupling bar and locking device, and (d) lateral openings. The method of unloading the containers is shown in *Fig.* 10, where (e) is the unloading platform, (f) a guide rail, (g) a winding rope and a winch, and (i) a measuring gauge. The lightly-constructed steel containers are rolled off the lorry by means of a rope and winch on to a platform. The capacity of each container is from 2½ to 3 tons. Special coupling bars and

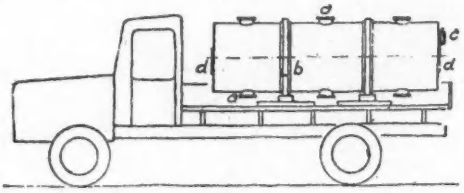

Fig. 9.

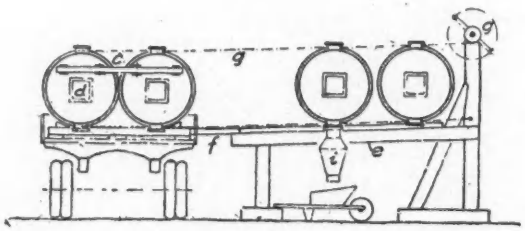

Fig. 10.

locking devices are provided at the sides of the lorries to secure the containers in position during transport, and large openings at the sides enable them to be cleaned by scraping.

It is reported in a recent number of "Revue des Materiaux" that about 70 per cent. of the cement produced in Western Germany is transported in bulk. In the United States, according to a report in "Cement" (Holland), about 40 per cent. of the cement used is transported in bulk. These journals also describe a Swedish steamship of 1,600 tons converted for this purpose. The ship can be loaded by one man in six hours and discharged by two men in twelve hours.

Proportioning and Control of Raw Mixtures.

VARIATION in composition of the raw mixture affects the operation of the kiln in that the ease with which the mixture can be burnt to a satisfactory clinker changes. For steady production and high fuel-efficiency accurate control of composition is important, since periods of easy burning do not compensate for loss in output and high fuel consumption during periods of hard burning. Also, variation in kiln-operation interferes with the attainment of uniformity in the product.

The methods of control depend to some extent upon the arrangement of the plant. The process used, however, generally is either continuous or in two stages.

Continuous Proportioning.

The raw materials move in separate streams towards a common point where they are combined into a single stream. This is the only proportioning operation, the remaining operations being concerned with the reduction of the mixture to a desired degree of fineness. Control of composition in a single proportioning operation is generally unsatisfactory. In order to remove variations in composition the usual practice is to fill silos or tanks consecutively and to draw from them simultaneously for the kiln feed. Thus the material drawn is a mixture of materials proportioned at widely different times. This constitutes a mechanical averaging tending to smooth out variations in composition.

Two-Stage Proportioning.

The first stage is a continuous proportioning process, supplying tanks of slurry to be proportioned in batches in the second stage. The materials should be proportioned roughly in the first stage, some tanks having higher and some lower than the required carbonate content. These are then combined in calculated proportions to form the final feed for the kiln. If the preliminary mixtures are fairly close to the desired composition, the degree of accuracy required in the second proportioning operation is not so important.

Mixtures of More Than Two Materials.

Where the control of the composition requires the use of more than two materials, accurate control cannot be maintained in a single operation. The two-stage process is the least that can achieve the required mixture. The continuous proportioning process in the first stage supplies tanks of slurry, which, after analysis, are proportioned in the second or subsequent stages in batches. Under these conditions potential composition is under direct control.

Mixtures of Two Materials.

For raw mixtures produced from two materials the common method of control consists of a determination of the calcium carbonate content. The limitations of this method of control should, however, be clearly realised. Cement raw materials are obtained from natural deposits which are somewhat variable in composition. If a reasonable control is to be kept on the C_3S-content, the proportions used in the mixture must also be changed. These changes are generally accompanied by changes in the carbonate content required to secure a satisfactory

composition. Changes in ignition loss and silica ratio of the raw materials are the main factors which lead to the necessity of changing the amount of carbonate.

Acid-Alkali Method of Control.

This is the method in most common use. It depends upon the decomposition of calcium carbonate by a measured quantity of standard acid in excess of that required by theory and then determining the excess acid by titration with standard alkali. The acid (hydrochloric) should be a two-fifths normal solution (1 c.c. = 0·02 gr. $CaCO_3$). The alkali (caustic soda) should be a one-fifth normal solution (1 c.c. = 0·5 c.c. standard acid). The indicator used is a 1 per cent. solution of phenolphthalein in 50 per cent. alcohol. If a 1 gr. sample is used for the determination, then 1 c.c. of standard alkali is equivalent to 1 per cent. calcium carbonate.

The method of determination is as follows: Exactly 1 gr. of slurry, dried at 105 to 110 deg. C., and ground to pass through a 100-mesh sieve, is weighed into a 250-c.c. conical flask. The powder is covered with distilled water, and 50 c.c. of the standard acid are added from a burette. The sides of the flask are washed down and the contents warmed on a hot plate for five minutes. The sides of the flask are again washed down with about 50 c.c. of hot distilled water and boiled for two minutes. Six drops of indicator are added, and the solution titrated with standard alkali to a distinct pink colour. During titration the end point may be obscured by insoluble material. Just before the end point is reached the insoluble matter flocculates and settles rapidly, leaving a clear solution in which the end point can be obtained exactly. If the acid and alkali are made up exactly to these strengths and exactly 1 gr. of dried slurry is used, then the percentage of $CaCO_3$ is 100 minus the number of cubic centimetres of standard alkali used.

The Calcimeter Method of Control.

This is an indirect gas method in which the calcium carbonate is decomposed by acid and the evolved carbon dioxide gas is collected in a suitable apparatus and measured. Since the carbon dioxide is proportional to the calcium carbonate, the percentage of carbonate can be calculated from the volume of carbon dioxide found. Forms of instruments (calcimeters) which have been employed in the cement industry are the Dietrich-Fruhling, the Faija, and the Bauber calcimeters. A full description and methods of use of these calcimeters are given in "Cement Chemists' and Works Managers' Handbook." The method was in common use in the industry at one time but has now been almost completely superseded by the more reliable acid-alkali method. The acid-alkali and the calcimeter methods both give values for the CaO content (calculated from the calcium carbonate found) which are low, since small percentages of CaO and NgO may be present in some form other than carbonate. In addition, some of the other constituents may have a small influence upon the value obtained by the acid-alkali method. Both these possible sources of error may be overcome in the main by standardising the acid with a slurry of known CaO and MgO content.

The Ignition Method of Control.

The dried slurry is prepared for analysis by heating at a temperature high enough to covert it to clinker. Burners and muffle furnaces commonly used for

ignitions and fusions in analytical work do not generally develop the temperatures required for clinkering samples of raw mixture. A suitable high-temperature furnace is one in which the heating elements are rods of carborundum, and such furnaces have normal working temperatures up to about 1450 deg. to 1500 deg. C. Full details of the methods of analysis for SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, and ignition loss are given in this journal, March to September, 1949. This method of control is necessary where compound composition control includes other compounds in addition to C_3S.

Method of Proportioning Slurries.

Calculation of the proportions in which tanks of slurry have to be mixed in order to obtain the desired carbonate content is not difficult. As an illustration of the method it is assumed that the control tests for two tanks of slurry are as given in the following and that the required carbonate is 77·5 per cent.

	Tank No. 1	Tank No. 2
Moisture (per cent.)	38·0	42·0
Calcium carbonate	80·5	75·5
Deviation from required carbonate ..	+ 3·0	+ 2·0

The deviations from the required calcium carbonate content, taken in the reverse order, represent the relative proportions of the two slurries, on a dry weight basis, required to obtain 77·5 per cent. carbonate in the mixture. That is, 2 parts

TABLE 1

FACTORS FOR CONVERTING WEIGHTS OF DRY SLURRY TO VOLUMES OF LIQUID SLURRY

Percentage of Moisture	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9
34·0	1·000	1·003	1·005	1·007	1·010	1·013	1·016	1·018	1·021	1·023
35·0	1·026	1·029	1·031	1·034	1·037	1·040	1·042	1·045	1·048	1·050
36·0	1·053	1·056	1·058	1·061	1·064	1·067	1·069	1·072	1·075	1·077
37·0	1·080	1·081	1·086	1·089	1·092	1·095	1·097	1·100	1·103	1·106
38·0	1·109	1·112	1·115	1·118	1·121	1·124	1·126	1·129	1·132	1·135
39·0	1·138	1·141	1·144	1·147	1·150	1·153	1·156	1·159	1·162	1·165
40·0	1·168	1·171	1·174	1·178	1·181	1·184	1·187	1·190	1·194	1·197
41·0	1·200	1·203	1·206	1·210	1·213	1·216	1·219	1·222	1·226	1·229
42·0	1·232	1·235	1·239	1·242	1·245	1·249	1·252	1·255	1·258	1·262
43·0	1·265	1·269	1·272	1·276	1·279	1·283	1·286	1·290	1·293	1·297
44·0	1·300	1·304	1·307	1·311	1·314	1·318	1·322	1·325	1·329	1·332
45·0	1·336	1·340	1·343	1·347	1·351	1·355	1·358	1·362	1·366	1·369
46·0	1·373	1·377	1·381	1·384	1·388	1·392	1·396	1·400	1·403	1·407
47·0	1·411	1·415	1·419	1·422	1·426	1·430	1·434	1·438	1·442	1·446

by weight of dry slurry in tank No. 1 and 3 parts by weight of dry slurry in tank No. 2 give the required composition. These may be converted to a relative slurry-volume basis by multiplying each of the relative weights by the moisture-factor obtained from *Table I*. Thus the relative volumes are :

	Relative weight (dry basis)		Factor (Table I)		Relative volume
Tank No. 1	2·0	×	1·109	=	2·218
Tank No. 2	3·0	×	1·232	=	3·696

The slurry tanks are generally of the same diameter, so that the proportioning is based on depths of slurry, the amount in feet being drawn in the required volumetric ratios. Thus if 30 ft. are drawn from tank No. 2 the amount to be drawn from tank No. 1 is $\frac{30 \times 2{\cdot}218}{3{\cdot}696} = 18$ ft.

The effect of errors in proportioning is proportional to the differences between the carbonate contents of the two tanks. Thus in the example an error of 1 per cent. in the proportions will cause an error of 0·05 per cent. in the carbonate content of the mixture. Because of the physical condition of slurries it is difficult to measure volumes with a high degree of accuracy, so that it is important that the contents of the tanks which are to be mixed should not deviate greatly from the desired composition.

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Japanese Views on Cement Formulæ and Phase Diagrams.

THE validity of some well-known formulæ for cement derived stoichiometrically, some formulæ derived from phase diagrams, and formulæ containing minor constituents are examined by Mr. K. Hashimoto of the Government Chemical Industrial Research Laboratory, Tokio, in "Pit and Quarry" recently, and the relation between these formulæ and phase diagrams is considered in relation to research recently undertaken in Japan.

Cement formulæ which generally state the greatest amount of lime that can combine with the acidic oxides during burning can be expressed in the general form $C = \sigma S + \alpha A + \phi F + \mu M + \gamma SO_3$, where the chemical symbols have the usual significance, that is C = CaO, S = SiO_2, A = Al_2O_3, F = Fe_2O_3, and M = MgO. The values of the coefficients σ, α, ϕ, μ, and γ for some of the formulæ propounded by well-known investigators are given in *Table* 1. In formulæ

TABLE I.

	$\frac{m}{n}$ C_mS_n σ	$\frac{m}{n}$ C_mA_n θ	C-A-F	$\frac{m}{n}$ C_mF_n Φ	MgO μ	$CaSO_4$ γ	Free CaO
1) Le Chatelier (1887)	3 2.8	3 1.65	.....	.....	—1.4	.....	.....
2) Newberry (1897)	3 2.8	2 1.1	.....	.....	.....	.....	.....
3) Kuhl (1914)	3 2.8	3 1.65	.....	3/2 0.54	.....	.....	.....
4) Hendrickx (1922)	2.5 2.33	3 1.65	(611)	3 1.05	.....	.....	.....
5) Merriman (1926)	3 2.8	3 1.64	(611)	3 1.05	.....	.....	.....
6) Nagai I (1929)	3 2.8	3 1.65	(511)	2 0.7	.....	0.7	.....
7) Janecke (1929)	2 1.86	4 2.2	(611)	2 0.7	.....	.....	+5%
8) Guttmann und Gille I	3 2.8	3 1.65	(411)	1 0.35	.....	0.7	.....
9) G & G II = Kuhl (1929)	3 2.8	2 1.1	(411)	2 0.7	.....	0.7	.....
10) Bogue = Nagai II	3 2.8	3 1.65	C_4AF	1 0.35	.....	0.7	.....
11) Lea and Parker (1935)	3 2.8	1.18	C_4AF	0.65	.....	.....	.....

1 to 9, which are derived from stoichiometrical relations, as is also formula(10), the coefficients in *Table* 1 are based on the following ratios :

$$\text{For silicates } (C_mS_n) : \sigma = \frac{m}{n} \cdot \frac{CaO}{SiO_2} = \frac{56}{60} \cdot \frac{m}{n} = 0{\cdot}93\frac{m}{n}.$$

$$\text{For aluminates—}(C_mA_n) : \alpha = \frac{m}{n} \cdot \frac{CaO}{Al_2O_3} = \frac{56}{102} \cdot \frac{m}{n} = 0{\cdot}55\frac{m}{n}.$$

$$\text{For ferrites—}(C_mF_n) : \phi = \frac{m}{n} \cdot \frac{CaO}{Fe_2O_3} = \frac{56}{160} \cdot \frac{m}{n} = 0{\cdot}34\frac{m}{n}.$$

$$\text{For magnesia : } \mu = \frac{CaO}{MgO} = \frac{56}{40} = 1{\cdot}4.$$

$$\text{For sulphate radical : } \gamma = \frac{CaO}{SO_3} = \frac{56}{80} = 0{\cdot}7.$$

In formula (10), where the Brownmillerite (C_4AF) is considered for the first time, the aluminoferrite is divided into the binary compounds, $C_4AF = C_2A + CF$, and the coeficients α and ϕ are derived for each aluminate and ferrite. Inversely, it can be said that Brownmillerite is already assumed in formulæ (8) and (9).

As the formulæ were not all proposed as cement formulæ, explanations are needed. Formula (1) was put forward by Le Chatelier[1] and is nearly correct, but μ is derived from the molecular ratio of CaO to MgO, is negative, and is not sufficiently justified.

S. B. and W. B. Newberry[2], formula (2), stated that a high alumina content is analogous to free lime in Portland cement, and they replaced the compound C_3A in Le Chatelier's formula by a hypothetical compound C_2A. Wetzel studied in 1913 the microscopical structure of clinker having the chemical composition $C_3S + S_2A$ and found it to be composed of only one crystalline phase in the ground mass. In Rankin's diagram of the ternary system $CaO - Al_2O_3 - SiO_2$, the line $C_3S - C_2A$ crosses the field of C_3S lengthwise, and in Germany this line was considered as the theoretical limit of soundness, which must not be overstepped in proportioning the raw materials. As the line $C_3A - C_3S$ lies entirely in the field of lime, Le Chatelier's formula is too severe, and the Newberrys' formula is more rational although based on a hypothetical compound.

Kühl's formula[3] assumed the compound C_3F_2, which was introduced by Hilpert and Kohlmeyer (1909). That $C_3A + C_3F$ gives C_6AF_2 was acknowledged by Janecke in 1928, Nagai-Sawayama in 1934, and more recently by Swayze (1946). The formula of Hendrickx[4] assumes a peculiar compound, $C_{2\cdot5}S$, which is not recognised by other investigators. Merriman's formula[5] is derived from the lime-silica index giving the greatest value 2·8 when all calcium silicate is C_3S. The first formula of Nagai[6] is derived from the lime-silica molecular ratio, using the highest value 3.

Janecke[7] showed that the best composition of Portland cement is when $\frac{S}{35} + \frac{A}{31} + \frac{F}{59}$ is 0·95, which allows the presence of 5 per cent. of free lime. Kühl has given this expression as $CaO = 1{\cdot}86SiO_2 + 2{\cdot}2Al_2O_3 + 0{\cdot}7Fe_2O_3$ + free lime, which assumes the compounds to be $xC_2S + yC_4A + zC_2F$ + free lime. Janecke did not agree with this, because the compound Janeckeit is referred to only indirectly as $2C_2S + C_4A = C_6AS_2$.

One of the formulæ of Guttman and Gille[8] applies when the ratio of Al_2O_3 to $Fe_2O_3 + Mn_2O_3$ is greater than 0·64, as in high-alumina and Ferrari cements. The second formula is for the ferritic cements and applies when the foregoing ratio is less than 0·64. Kühl claims to have proposed this formula previously. Although the coefficients are the same in Bogue's formula[9] as in Guttman and Gille's first formula, the ternary compound C_4AF is also mentioned.

It is evident that the ratio of m to n in the compounds C_mS_n, C_mA_n, and C_mF_n must be the highest of all the values established by experiment, and from this viewpoint the value of $\frac{m}{n}$ is too low for C_2A in formulæ (2) and (9), for C_3F_2 in formula (3), for $C_{2\cdot5}S$ in formula (4), for C_2S in formula (7), and for CF in formula (8). In formulæ (4) and (5), $\frac{m}{n}$ for C_3F is too large. The compounds C_4A and C_6AS_2 in formula (7) do not actually exist.

There remain only formulæ (6) and (10) which are the basis of the Nagai-Bogue calculation in which stoichiometrical relations only are involved, and not the phase diagram. As already mentioned, the connecting line C_3S–C_3A lies entirely in the field of lime. For a product such as Portland cement, equilibrium is hardly attained during cooling, especially in the range of the solid phases, so that resorption of free lime is scarcely realised. This is the reason why the lime-saturation degree of Kühl, $\frac{CaO}{2{\cdot}8SiO_2 + 1{\cdot}65Al_2O_3 + 0{\cdot}7Fe_2O_3}$, which is correct in view of compound association, usually gives lower values than in reality, and necessitates larger coefficients, such as those proposed by Hess[10] and Spindel[11].

Formula derived from Phase Diagrams.

Lea and Parker's formula is based on stoichiometrical relations for C_3S and on the phase diagram for α. Later[13] Lea showed that his formula, proposed with Parker, represents a plane in the tetrahedron of CaO–Al_2O_3–Fe_2O_3–SiO_2 and is based on three points: (1) Composition point for C_3S; (2) Composition point for C_4AF; and (3) Invariant point for CaO–C_3S–C_3A. However it is more desirable that other known points on the lime-saturation face be used, and in Lea and Parker's diagram the points are

		CaO	SiO_2	Al_2O_3	Fe_2O_3
K	CaO-C_3S-C_2S	68·4	22·4	9·2	—
D	CaO-C_3S-C_3A	59·7	7·5	32·8	—
T_1	CaO-C_3S-C_3A-C_4AF	55·0	5·8	22·7	16·5
X	CaO-C_3S-C_4AF..	52·8	5·6	16·2	25·4

As three points determine a plane, the following formulæ can be derived:

D-K-T_1: $CaO = 2{\cdot}55SiO_2 + 1{\cdot}24Al_2O_3 + 0{\cdot}74Fe_2O_3$.

D-K-X: $CaO = 2{\cdot}54SiO_2 + 1{\cdot}25Al_2O_3 + 0{\cdot}73Fe_2O_3$.

As both planes have almost the same coefficients, it is evident that the lime-saturation degree is expressed by only one formula without regard to the magnitude of the iron modulus. For this reason it is not right to have two formulæ for lime saturation. Since the line D-K crosses the field of C_2S, which is lower in lime content than C_3S, these two formulæ are both insufficient. Therefore it is better to adopt the composition point of C_3S instead of the point K.

As regards the quaternary invariant point, new data are available. Swayze[14] gives for 1342 deg. C. the following: SiO_2, 5·8; CaO, 53·9; Al_2O_3, 21·2; and Fe_2O_3, 19·1. Using these data and the two points C_3S and D, a plane passing through the three points is derived and gives the cement formula

$$CaO = 2{\cdot}8SiO_2 + 1{\cdot}18Al_2O_3 + 0{\cdot}66Fe_2O_3 \quad .. \quad .. \quad .. \quad .. \quad (i)$$

This is nearly the same formula as that of Lea and Parker, and the plane represented thereby is the triangle shown boldly in *Fig.* 1.

Formulæ Including Minor Constituents.

Lea has given another formula which includes magnesia instead of iron oxide, namely:

$$CaO = 2{\cdot}8SiO_2 + 1{\cdot}18Al_2O_3 - 0{\cdot}93MgO \quad .. \quad .. \quad .. \quad .. \quad .. \quad (ii)$$

This formula represents the plane shown in *Fig.* 2 as a tetragon. Formulæ (i) and (ii) can be combined to give

$$CaO = 2{\cdot}8SiO_2 + 1{\cdot}18Al_2O_3 + 0{\cdot}66Fe_2O_3 - 0{\cdot}93MgO.$$

It is also possible to combine the triangle and the tetragon. A method of graphical representation of a five-component system has been proposed by R. Kimura.[15] A regular pentahedroid is considered in four-dimensional space, and a projection is made on to a plane. By making the projection a regular pentagon the system can be represented fully symmetrically. The tetragonal pyramid shown in *Fig.* 3 is the section of the pentahedroid and represents the lime-saturation front, that is a complex-plane in the four-dimensional space.

Other components such as sodium and potassium can be considered on this basis, the coefficients for Na_2O and K_2O being $-0{\cdot}32$ and $-0{\cdot}61$ respectively. No phase diagram is available for TiO_2 and Mn_2O_3, but titanium oxide seems to have a coefficient of nearly the same order as that of silica, and manganese oxide seems to replace iron oxide.

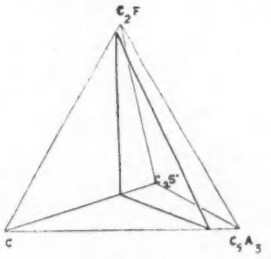

Fig. 1.

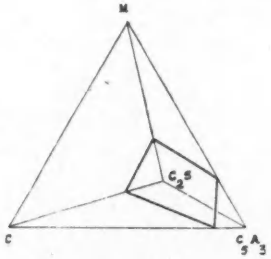

Fig. 2.

Allowing corrections for gypsum and free lime, the cement formula now becomes

$$CaO - 0{\cdot}7SO_3 - \text{free lime} = 2{\cdot}8SiO_2 + 1{\cdot}18Al_2O_3 + 0{\cdot}66Fe_2O_3 - 0{\cdot}93MgO - 0{\cdot}32Na_2O - 0{\cdot}61K_2O.$$

The lime degree is the ratio of the left-hand side of this equation to the right-hand side, and was calculated by this expression for several samples of cements that were analysed. Although the cements were not designated as rapid-hardening, there were some for which the value of the lime-saturation degree exceeded unity. This is probably accounted for by the existence of free lime, which was not determined. However, since the cement formula is a linear approximation to the lime-saturation front, unity may in some cases be exceeded.

Remarks.

The Swayze diagram is based on the fact that C_2F is a congruent melting compound, as was first pointed out by Yamauchi,[16] who prepared pure C_2F and observed on heating it to 1470 deg. C. that there occurs neither dissociation nor transformation. Yamauchi also extended the solid-solution range of the

iron-bearing phase beyond C_4AF to C_2F. For the extreme member of the solid-solution series he gave

$$5C_2F.3C_6A_3.2C_3A = C_{6\cdot2}A_{2\cdot2}F$$

instead of C_6A_2F, which he also prepared but regarded as merely a member of the series. For the four components CaO, SiO_2, Al_2O_3, and Fe_2O_3 it is better, as Kühl has said, to replace the hydraulic modulus by the lime-saturation degree, but, when a fifth component is considered, four co-ordinates are needed to fix the point. For this purpose the lime-saturation degree, together with the hydraulic modulus, the silicate modulus, and the iron modulus, can be used. Inclusion of

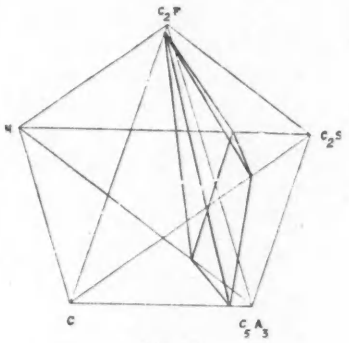

Fig. 3.

one or two other components necessitates the introduction of additional ratios, which can be chosen arbitrarily. The following may be considered: Potash ratio $= \frac{M}{K+N}$; Soda ratio $= \frac{M}{N}$ or $\frac{K}{N}$.

Many investigations have been made to determine the most favourable composition of high-alumina cements, but Kühl concludes that there is no single valid compositional formula, because the effective compounds C_5A_3, CA, C_3A_5, and C_2S have no common front on which the best quality is expected, so that a single plane cannot be fitted thereon.

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Methods of Determining the Insoluble Residue of Cement.

IN the Bulletin of the Roumanian National Institute of Technological Research (Volume 3, 1948, Nos. 3 and 4), Mr. A. Steopoe describes a modified method for determining the amount of matter in Portland cement insoluble in hydrochloric acid. Instead of boiling the residue in a 5 per cent. solution of sodium-carbonate, the author recommends washing the residue with a cold 2 per cent. solution of sodium-hydroxide, an operation which is carried out with the same filter and which gives results practically the same as those by other methods.

The German Method.

Hitherto the German method has been used in Roumania, in accordance with which 2 g. of cement are placed in a 400-cc. beaker with 100 cc. of water into which is then mixed 50 cc. of 50 per cent. hydrochloric acid. The mixtures are warmed rapidly until they just become clear, filtered immediately, and washed with boiling water. The filter paper containing the residue is placed in a glass where it is treated with 50 cc. of 5 per cent. solution of sodium-carbonate, is torn into shreds with a rod, and is then boiled to re-dissolve the silica that has separated. It is filtered while boiling. The alkaline salts are washed out with boiling water, then with very dilute hydrochloric acid, and again with boiling water. The residue and the filter are dried and burnt.

The German method requires large quantities of the reagents and of filter paper. It has been shown that to decompose completely 1 g. of cement containing 67 per cent. of CaO, 7 per cent of Al_2O_3, 3 per cent. of Fe_2O_3, and 2 per cent. of MgO, and to transform these constituents into chlorides, 2·5 cc. of concentrated (1·19) hydrochloric acid (and consequently 5 cc. of 50 per cent. hydrochloric acid) only are necessary, whereas the amount used in the German method is five times this quantity. The operations are also complicated, and two filters are necessary for a single determination. Another disadvantage is the lack of precision in the duration of the boiling with the solution of sodium-carbonate, an important matter in the case of cements in which trass or other admixtures are present. The author has shown elsewhere that the solubility of the silica, which reacts when boiled with alkaline solutions, varies greatly with the concentration of the alkaline solution, the duration of the process, the temperature, and the method of making the test. Consequently the same cement with an admixture of trass gives different results dependent upon the conditions of the test. For this and other reasons, it is proposed that the alkaline washing of the residue of the cement decomposed by hydrochloric acid must be made when cold, and that instead of a 5 per cent. sodium-carbonate solution, which when cold does not dissolve all the coagulated silica, a 2 per cent. solution of NaOH should be used. Experience shows that the same filter can be used throughout.

Roumanian Method.

The Roumanian method, which is to be included in the new Roumanian standards for Portland cement, is to weigh precisely 1 g. of cement and place it in a

200-cc. Berzelius beaker. Add 25 cc. of water and decompose the suspension by adding drop by drop, while shaking, 10 cc. of concentrated hydrochloric acid. The reaction is hastened by breaking lumps of cement by a glass rod with a flattened end. When all the cement has decomposed the suspension is heated rapidly to bring it nearly to boiling in a short time. If the cement is pure the solution will clarify and pass from a clear yellow to an orange hue. The suspension is filtered on a white filter, which is then washed five times with boiling water. There remains on the filter the insoluble substance in the cement and very little of the silica precipitated during heating. To remove the residue the filter paper, while still in the funnel, is washed four or five times with 100 cc. of cold 2 per cent. solution of NaOH, then in succession twice with boiling water, twice with 0·7 per cent. hydrochloric acid, and five times with boiling water. The wet filter is torn into shreds and dried in a crucible, and is then burnt to constant weight. The weight of the residue obtained multiplied by 100 gives the percentage of insoluble matter in the cement.

The German and Roumanian methods were applied to samples of seven brands of ordinary Portland cement and five brands of high-strength Portland cement. In only one case (a high-strength cement) were the results identical; in two cases the Roumanian method gave results up to 0·02 per cent. less than the German method; but in the remaining cases the Roumanian method gave greater results, the greatest difference being 0·06 per cent., which is within the range of experimental errors. (A description of a British method of determining the amount of insoluble residue was given on page 78 of this journal, July, 1949).

Composition of Magnesian Chloride Cement.

Standards for Sorel cement were issued in 1943 in Germany (D.I.N. 272, 273) and some notes on these are given by Sr. P. L. Gomez in "Cemento-Hormigon," June, 1948. In his original investigation Sorel stated that the ratio of MgO to Cl_2Mg was 5 : 1. Other investigators have either confirmed this ratio or have postulated other values, and in particular have given the proportion of water. For example, in 1932 Lukens (as quoted by Lea and Desch) found the ratio of the three constituents MgO, $MgCl_2$, and H_2O to be 3 : 1 : 11. In "Zement" in 1937, the results obtained by several investigators were given; these include: 1 : 1 : 16 (André), 2 : 1 : 9 (Kallauner), 3 : 1 : 12 (Hundshagen), 5 : 1 : 13 (Davis), 5 : 1 : 14 (Hoff), 5 : 1 : 17 (Beuder), and 10 : 1 : 18 (Krausse). Sr. Gomez comments on the theoretical nature of these formulæ, and suggests that for practical purposes the expression should be 3 MgO : 1 $MgCl_2$: 10 H_2O. The quantity of water is of great importance as upon it depends the strength of the cement. The reactions commence about two hours after mixing and finish a variable number of hours later. Any excess of magnesia acts as an active material and produces visible expansion.

Cement Production of the World.

IN a special number of *Cemento Hormigon* for October, 1949, entitled "Informacion Mundial de la Industria del Cemento" (obtainable, price 25 pesetas, from Calle de Gerona, 69, Barcelona (9), Spain), data are given relating to the cement industry in various countries. The annual production is given for the years 1929, 1938, 1945, 1946, 1947, and 1948 for each country in Western Europe (excluding Eire); Asia (excluding Russia); Africa; North, Central and South America; and Oceana. The total production for the parts of the world mentioned was about 68,000,000 tons in 1929 and about 85,000,000 tons in 1948. The production in most countries in Eastern Europe and Russia is given completely only for the years 1929 (7,250,000 tons) and 1938 (11,500,000 tons). For most countries there are also given some notes on the industry, the types of cements manufactured, a list of the cement works, and notes on exports and imports. Maps show the positions of most of the cement works in the world.

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